

INKJET METROLOGY AND STANDARDS FOR ION MOBILITY SPECTROMETRY

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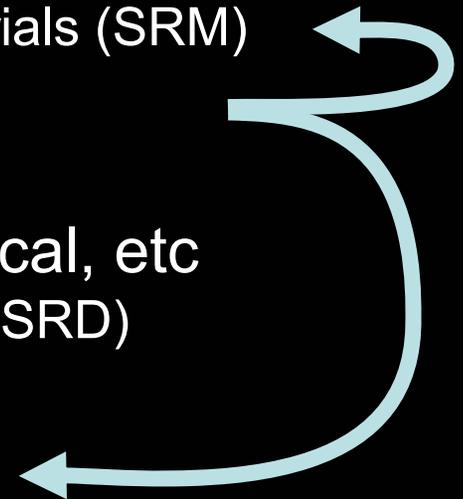


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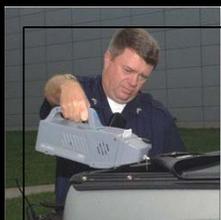
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Types of Standards

- Standard materials
 - chemical, physical, optical, etc
 - certified reference materials (SRM)
 - testbed (RM, IM, TM)
- Standard data
 - chemical, physical, optical, etc
 - certified reference data (SRD)
 - interactive (RD, MTD)
- Calibration services
- Documentary standards
 - performance or design
 - voluntary or regulatory



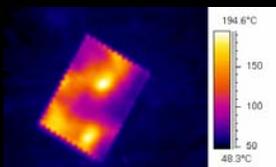
Trace Contraband Detection Using Ion Mobility Spectrometry



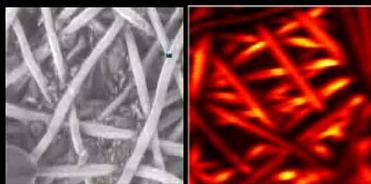
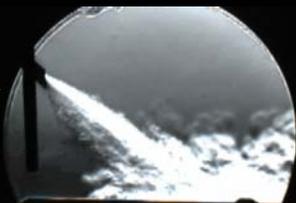
Vapor-based detection



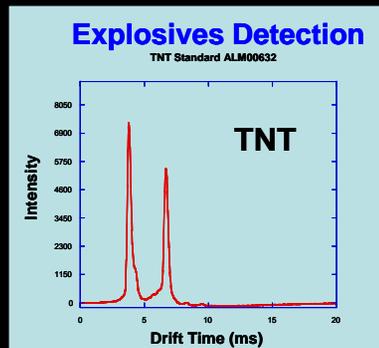
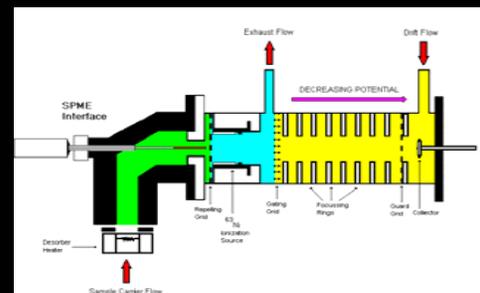
Swipe-based residue detection



Portal-based particle detection



Ion Mobility Spectrometer



Metrological Issues

- Massive deployment of IMS detectors
- Diversity of applied technologies
- Metrics of reliability & comparison of performance
- Consensus for technical improvements
- Prioritization of R&D
- Liability



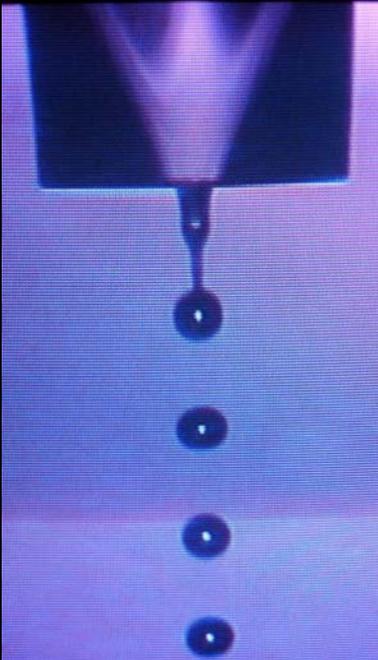
“Inkjet” Technology for Trace Contraband Metrology



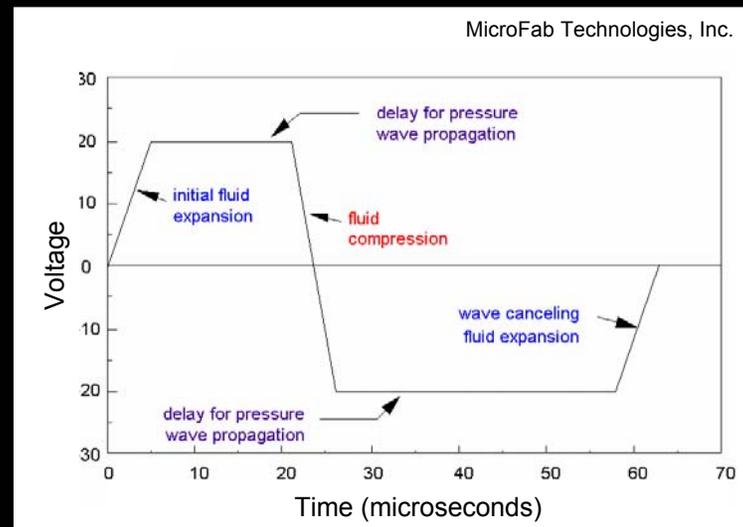
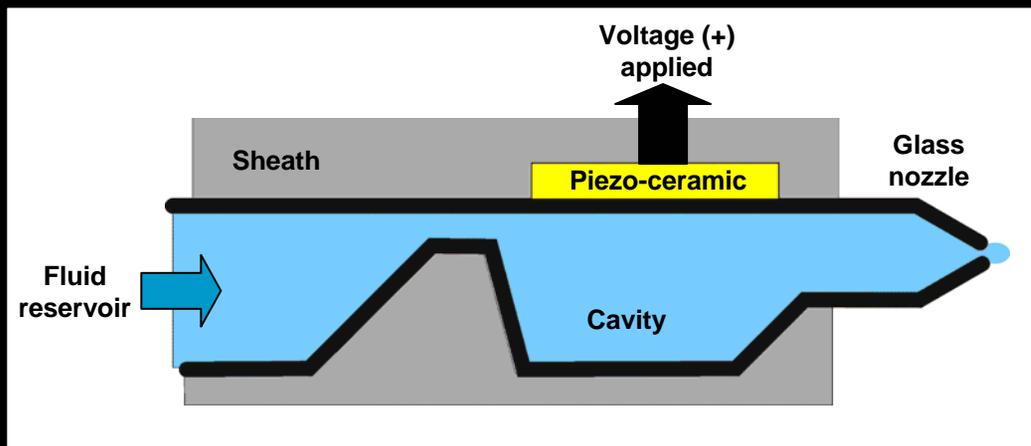
MicroFab Technologies, Inc.

Advantages

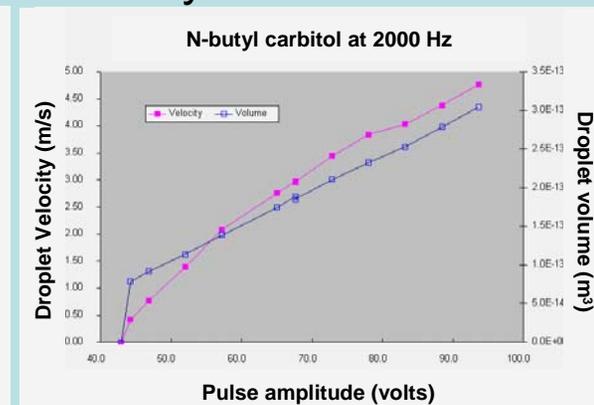
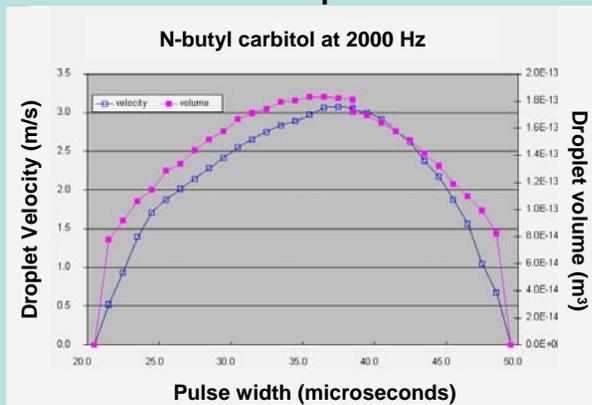
- Dispenses small quantities (picoliter-sized droplets, femtograms of analytes)
- High precision delivery
- Dynamic range (6+ orders of magnitude)
- Digital programmability
- Fieldability



Microdroplet Formation



Droplet Volume & Velocity Trends



MicroFab Technologies, Inc.

Piezoelectric Microdispensing

Technical Factors

Fluid factors

- Thermodynamics (viscosity, specific gravity, surface tension, vapor saturation pressure, heat capacity, boiling point), solute concentration, particles

Nozzle factors

- Piezoelectric waveform, cavity & orifice dimensions (droplet size, resonance limits: drop-on-demand), wettability (jet direction), fluid back pressure

Post-injection factors

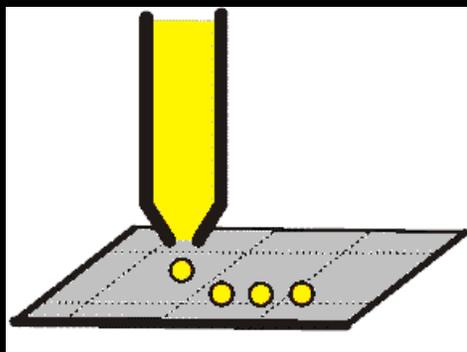
- Ambient media & flow rate, target surface characteristics (temperature, conformation, roughness)



Outline

- Microdispensing systems

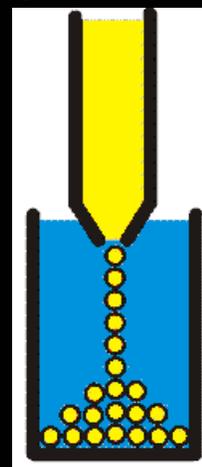
Arrays



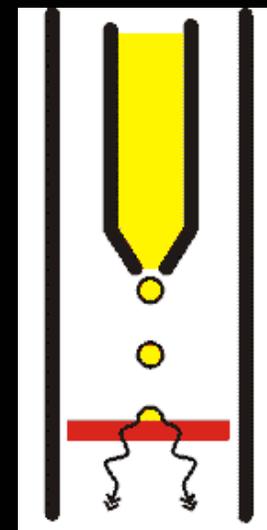
Particles



Spheres

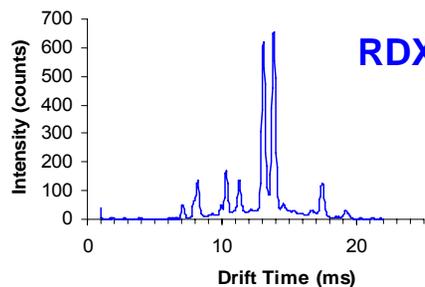


Vapors



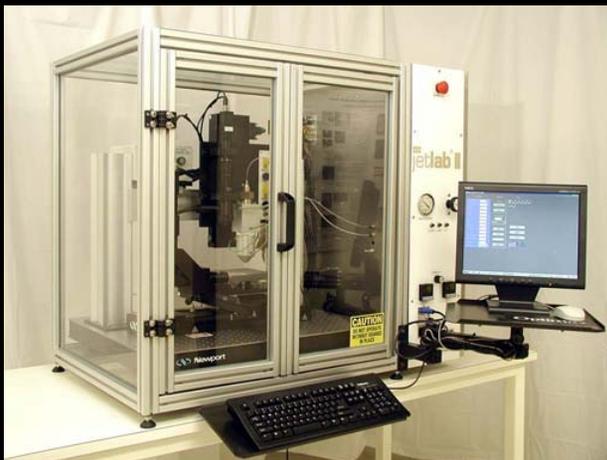
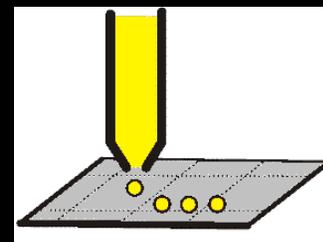
- IMS measurements

IMS Spectra of Sphere Test Sample

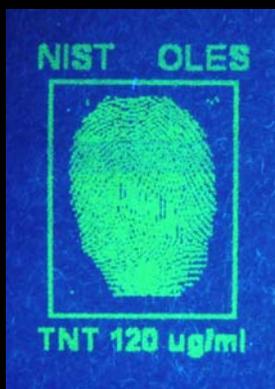


Quant-Jet

(MicroFab Technologies JetLab III)



- 2-D arrays & patterns
- IMs and TMs
- Verification
 - Optical
 - Microanalysis
 - IMS
 - Classical methods



1 mm

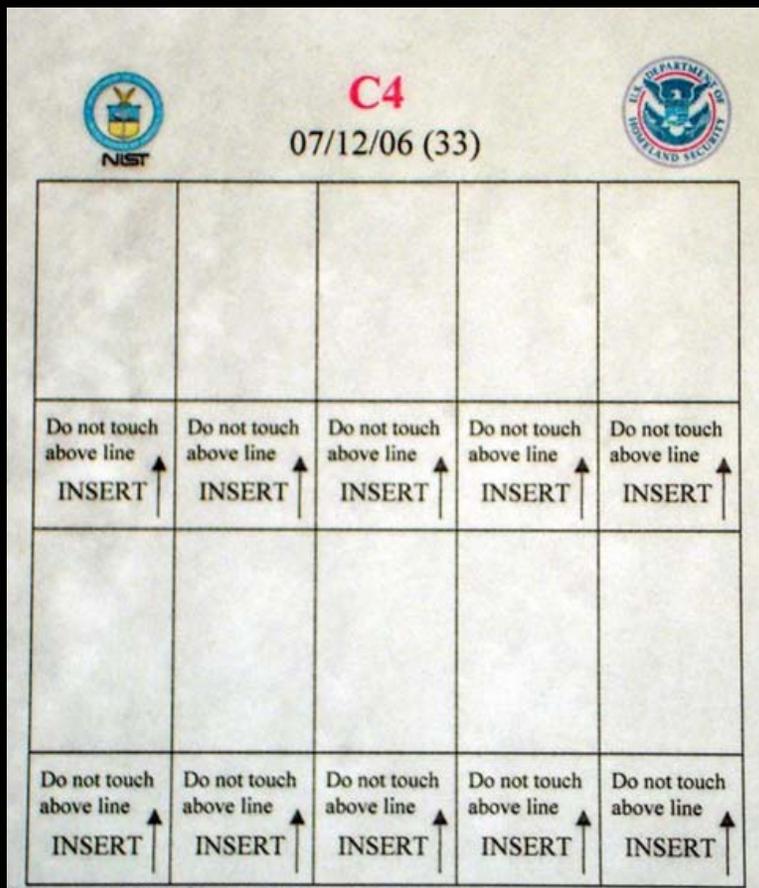
C4 + fluorescein on PTFE



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IMS Intercomparison Reference Material

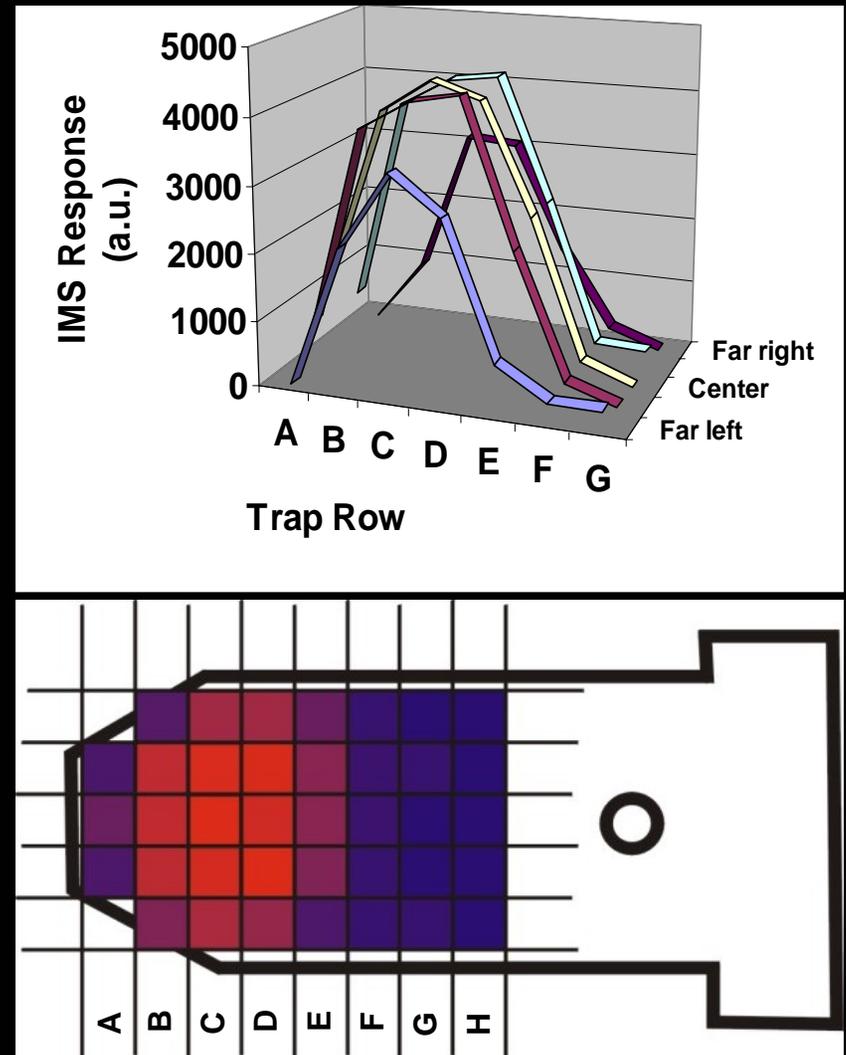


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Thermal Desorption

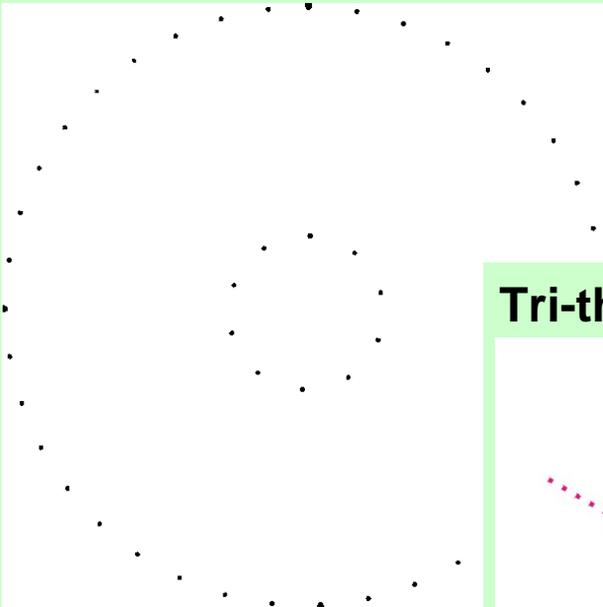
Analyte Position Factor on Sample Trap

- Aliquots of RDX solution placed on traps within virtual grid
- IMS measurements performed and replicated
- IMS response dependent on grid position
- Asymmetries indicated



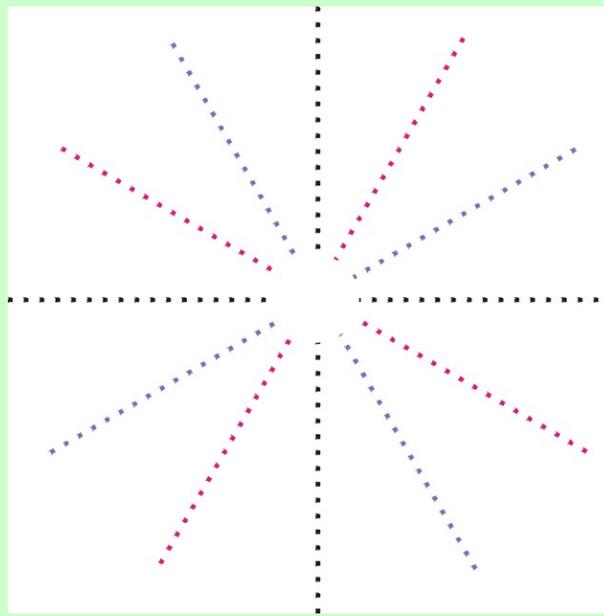
Thermochromic Inks

Concentric circles

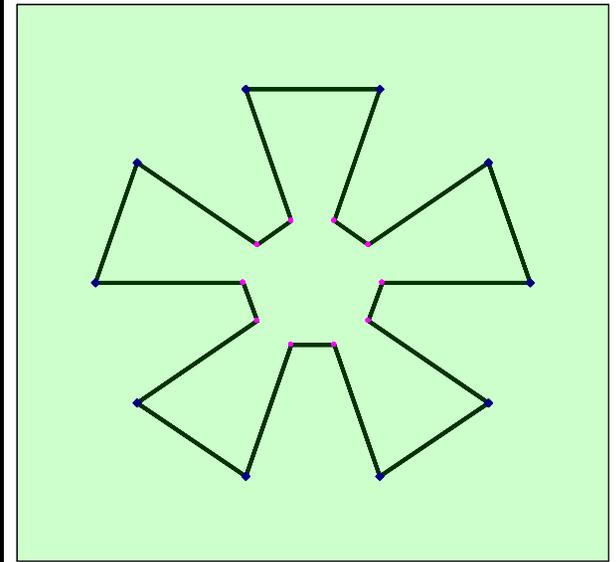


- Formulated for drop-on-demand inkjet printing
- Color change at specified temperature
 - Desorption temperature profile
 - Misuse of printed standard materials

Tri-thermochromic Radial

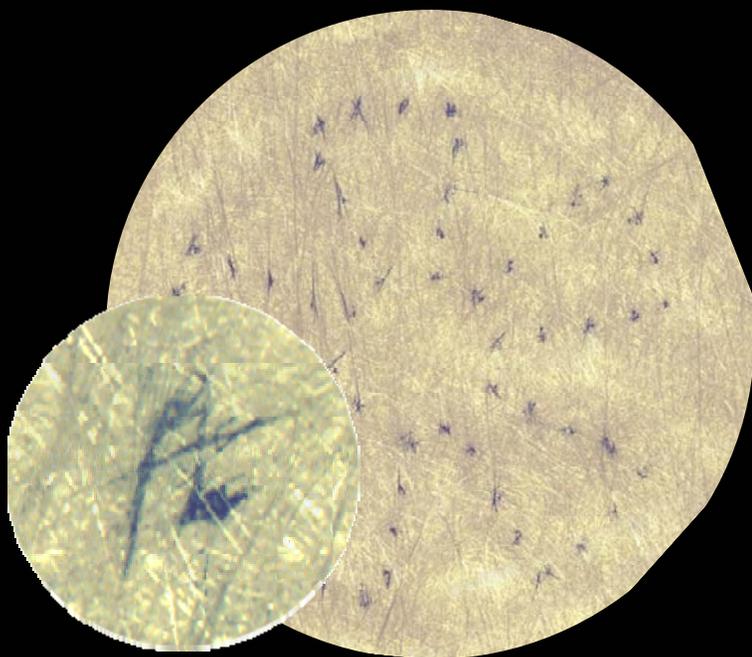


Gumby Cross

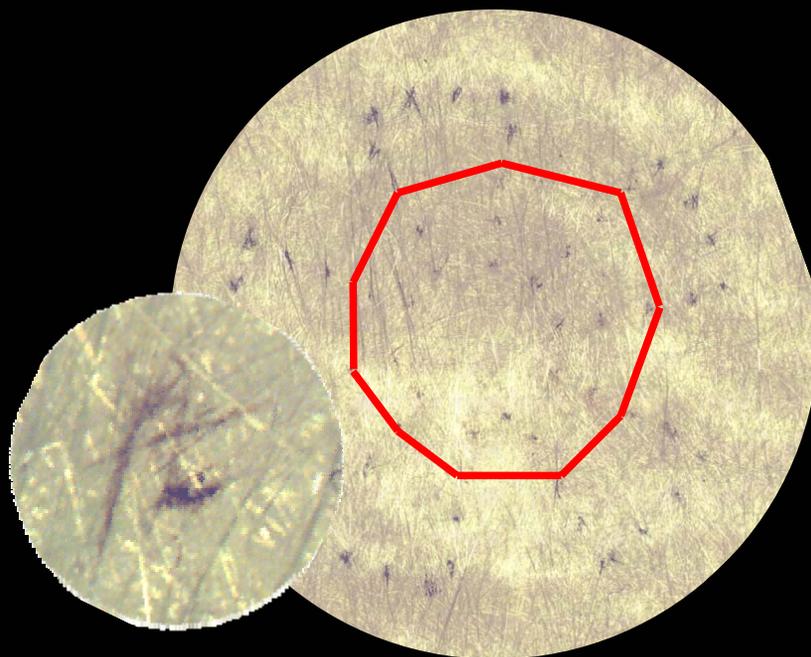


Thermochromic Indicator of Desorption Isotherm

PTFE swipe in Barringer IonScan 400B



Before desorption

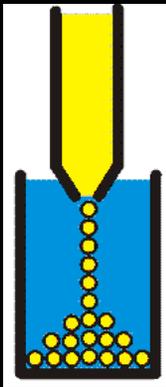


After desorption, 200 °C for
10 seconds



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Polymer Microsphere Formation using Emulsion/Solvent Extraction Printing “Sphere-Jet”



Pharmaceutical delivery technology applied to generation of IMS particle standards

- Non-toxic
- Biodegradable
- Monodisperse

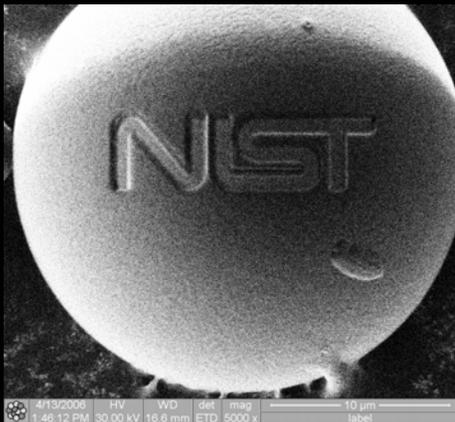


movie

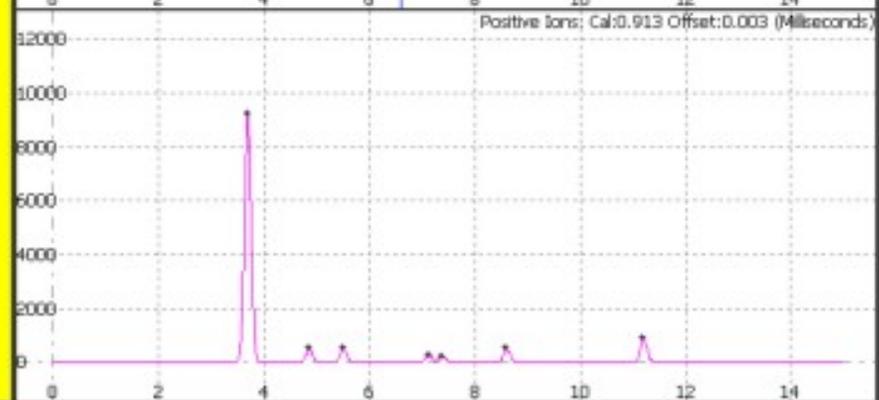
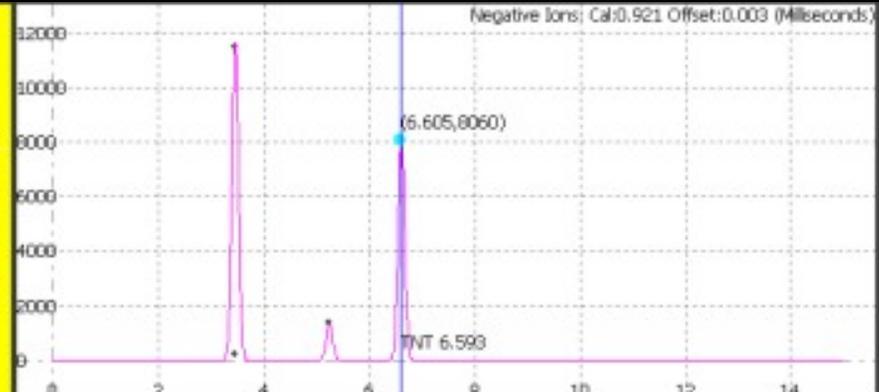


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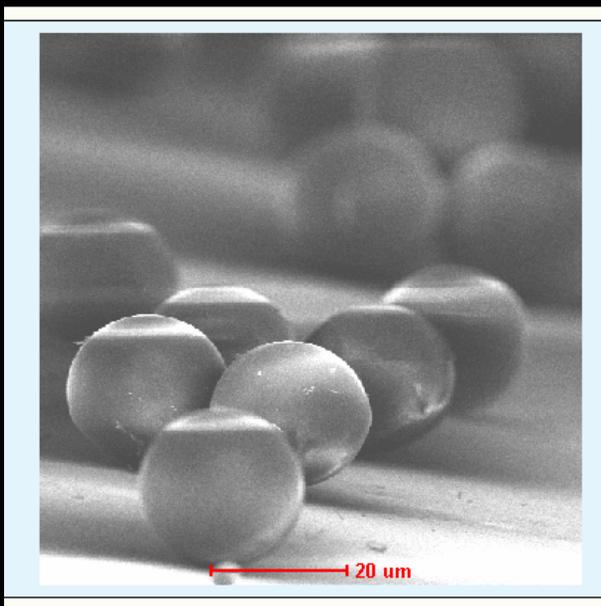
Polylactic-glycolic Acid (PLGA) – TNT Microspheres



Substances Detected			
Substance	Time	Strength	
TNT+	6.593	10.35	
Neg Ion Peaks		Pos Ion Peaks	
Time	Height	Time	Height
3.450	11455	3.690	9189
3.460	182	4.856	467
5.240	1337	5.504	494
6.605	8060	7.126	240
		7.390	198
		8.606	477
		11.207	870

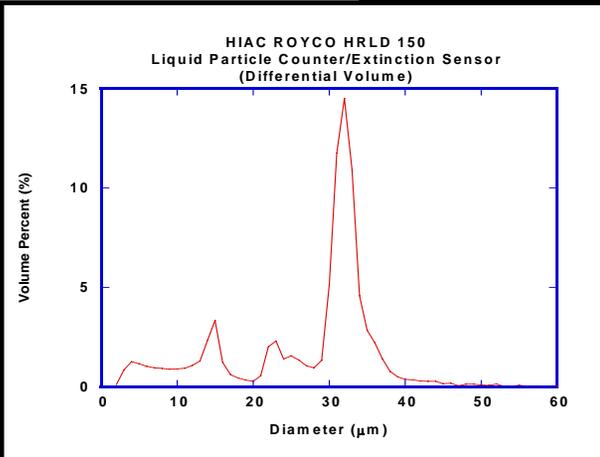
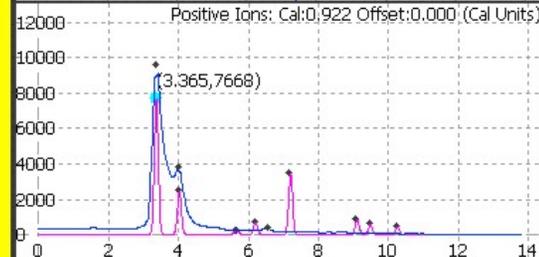
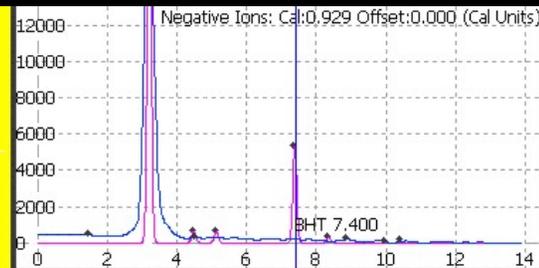


PLGA Microspheres containing Butylated Hydroxytoluene (BHT)

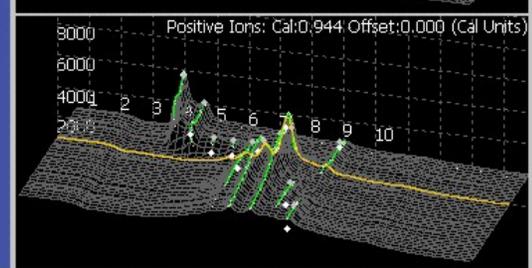
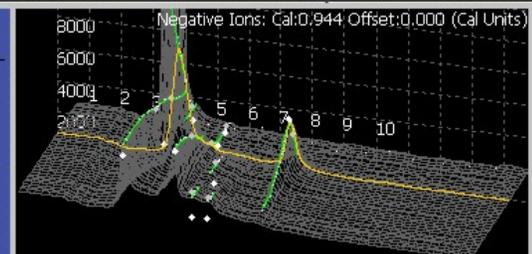


Substances Detected			
Substance	Time	Strength	
BHT+	7.400	6.22	

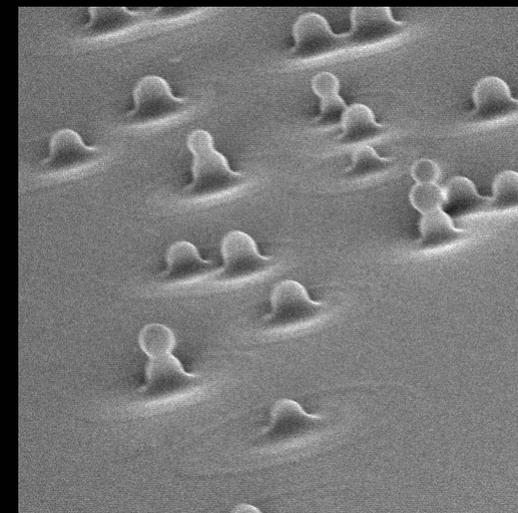
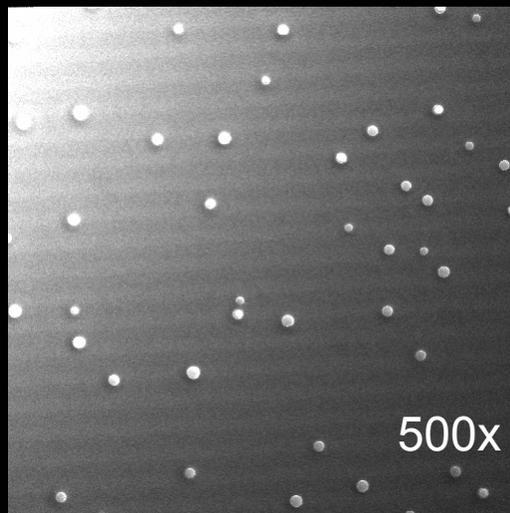
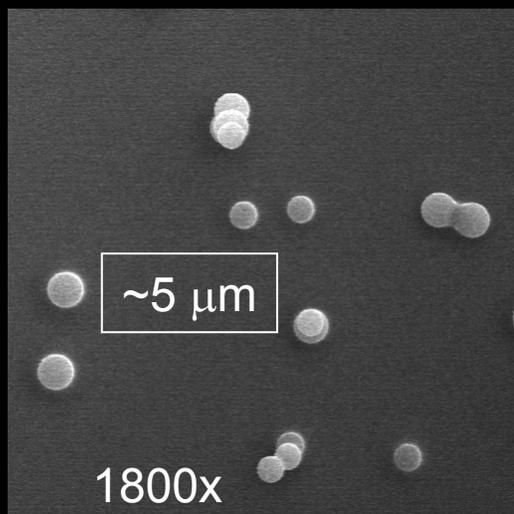
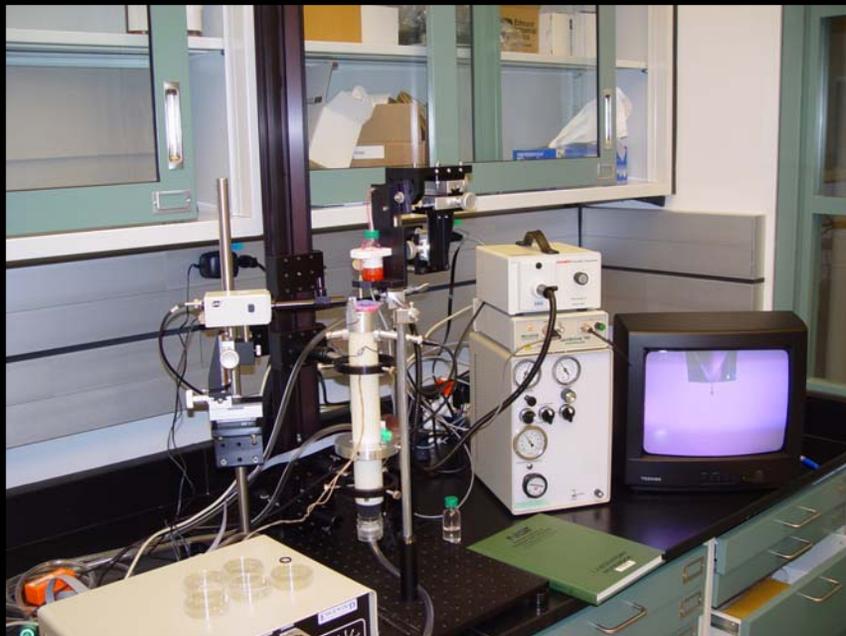
Neg Ion Peaks		Pos Ion Peaks	
Time	Height	Time	Height
3.185	20512	3.365	7668
4.466	628	4.024	2444
5.117	593	5.633	187
7.379	5323	5.688	137
8.352	290	6.203	664
		7.205	3454
		9.110	813
		9.484	547
		10.259	440



Neg Ion Peaks		Pos Ion Peaks	
Time	Height	Time	Height
3.177	16246	3.366	3630
3.259	3597	3.997	1099
3.700	537	4.694	284
3.812	2152	5.343	358
4.801	3976	5.892	707
4.809	342	6.528	2009
4.820	315	6.536	311
5.414	203	7.214	2950
5.421	201	7.915	413
5.896	292	8.396	401
7.340	3459	8.437	581



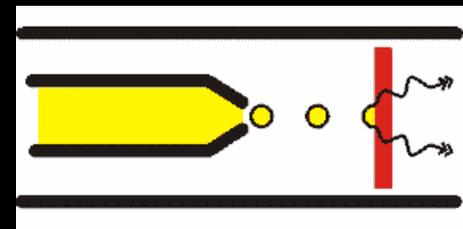
Particle-Jet



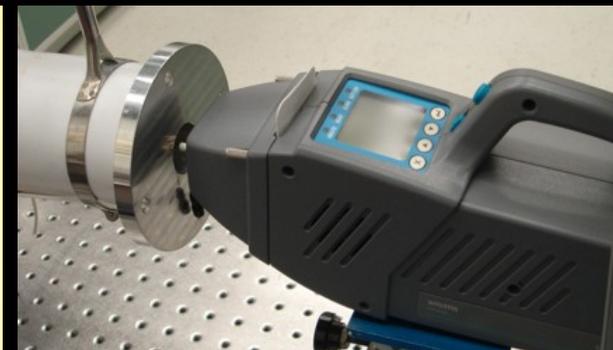
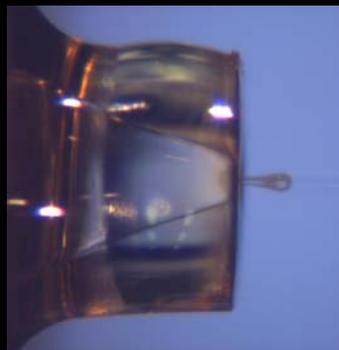
SEM Images of SEMTEX Particles



Vapor-Jet

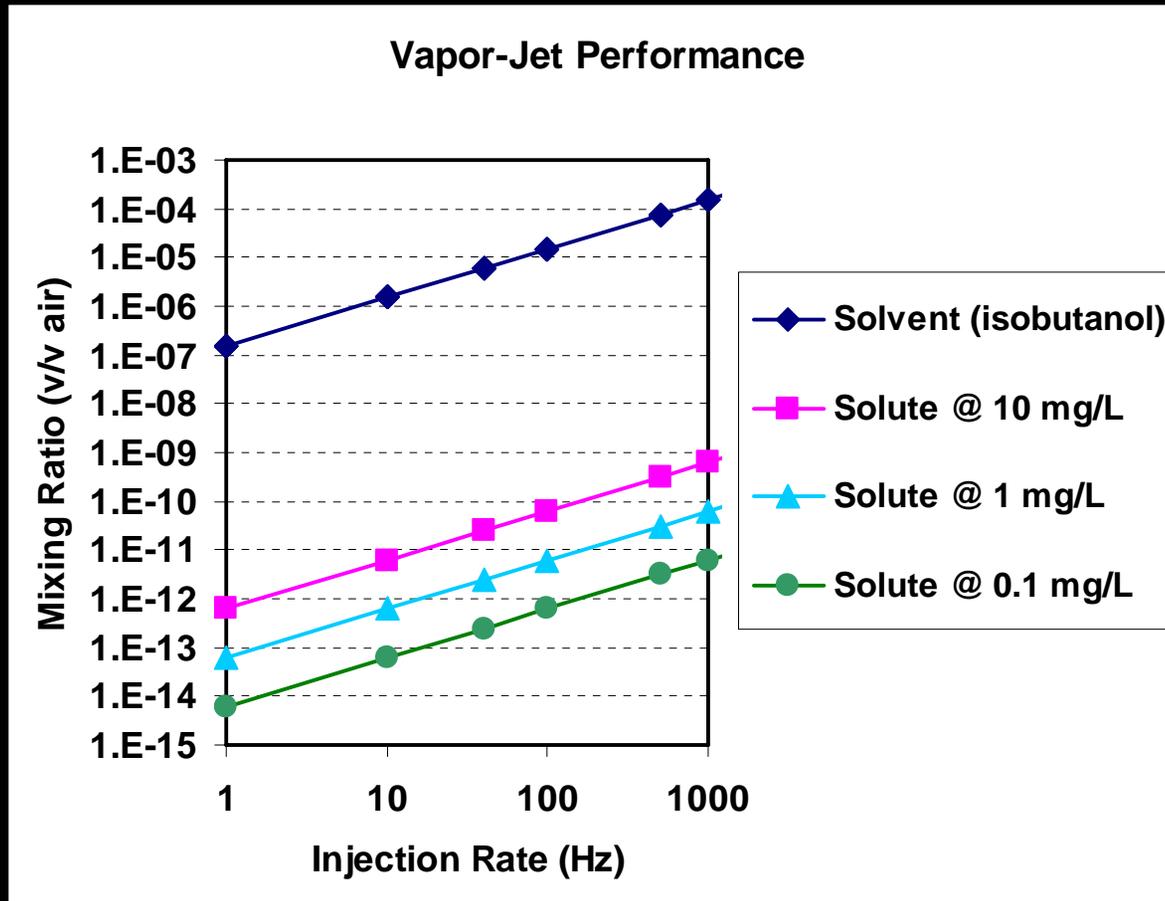


- Trace vapors of explosives, CWA simulants, odor signatures
- Calibration of vapor detectors
- Sets performance targets of next generation vapor detection technologies



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Designed Performance



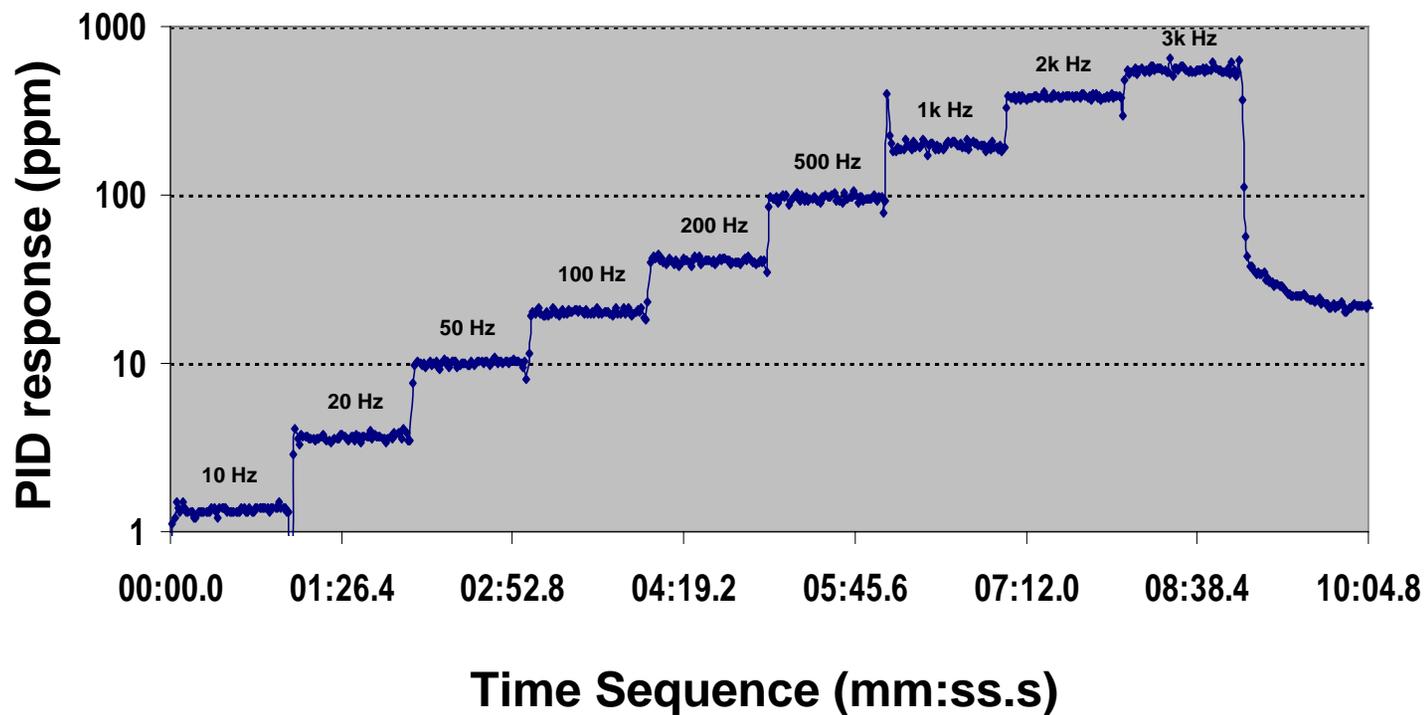
Microdroplet diameter = 58.4 micrometers
Air flow = 10 L/m



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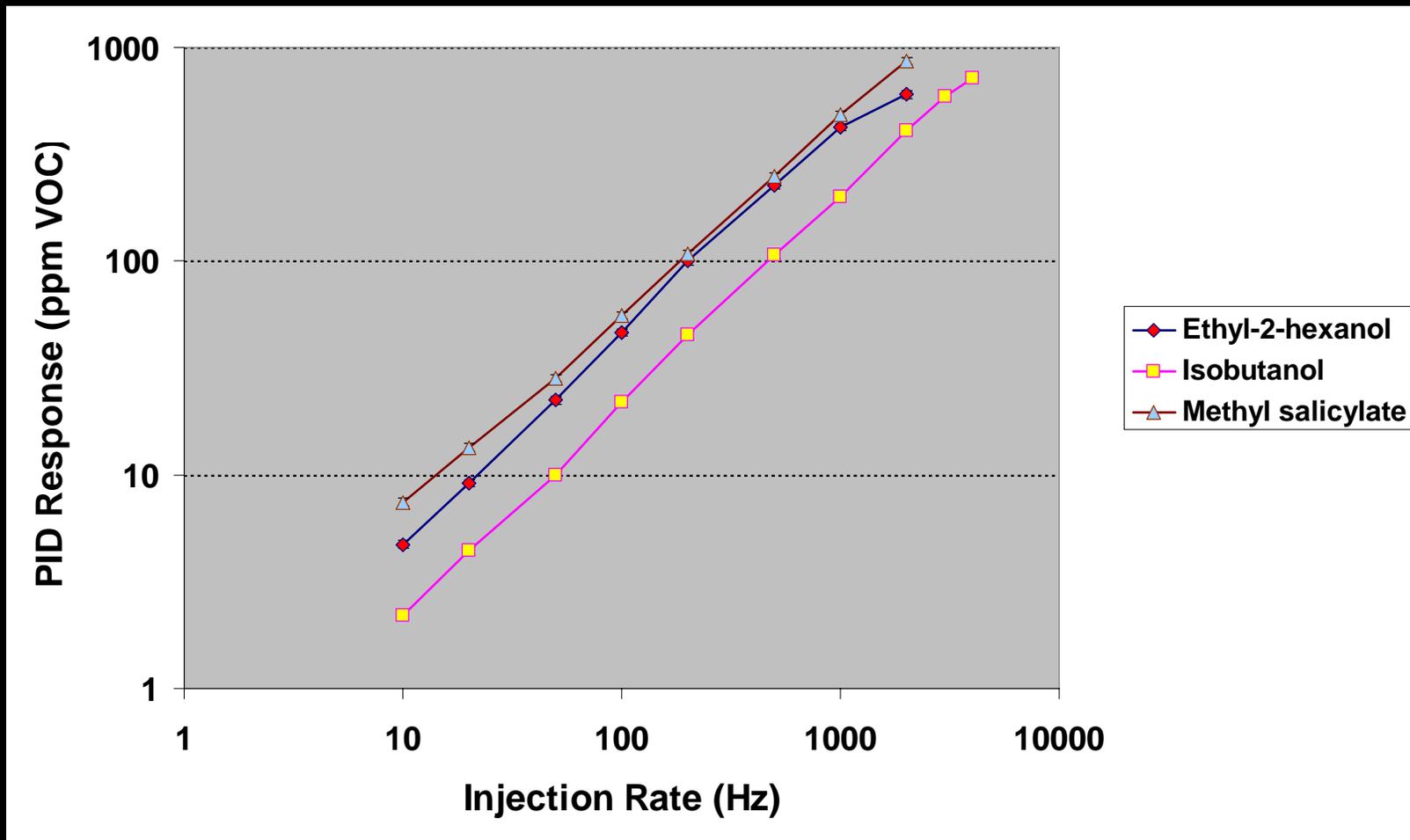
Photoionization Measurements

Isobutanol



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PID Results for 3 Fluids





Thermodynamics of Trace Solute Evaporation

Maximum Evaporative Mass Flux

$$\Gamma = (m \cdot p_s) / (2 \cdot \pi \cdot m \cdot k_B \cdot T)^{1/2}$$

Mass Injection Rate

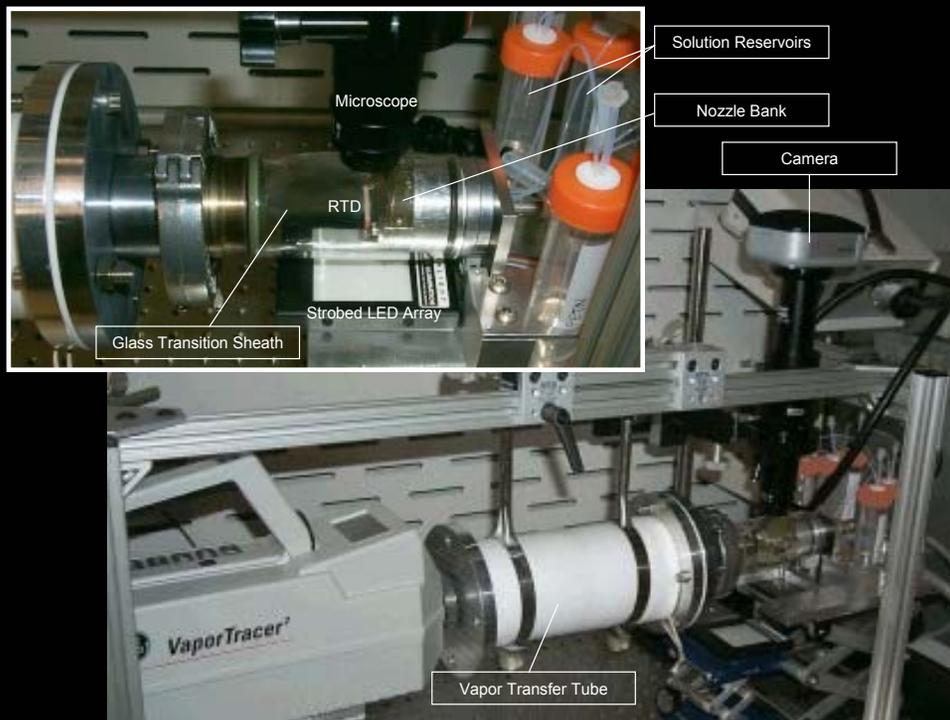
$$\Lambda = C \cdot v \cdot l$$

Table II. Coefficients for the Reduced Clausius-Clapeyron Equation (Eq. 2), and Calculated Evaporation and Injection Rates.

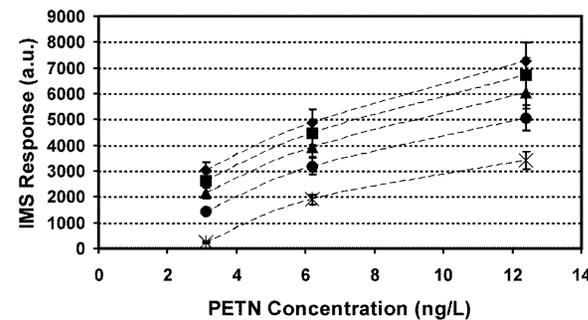
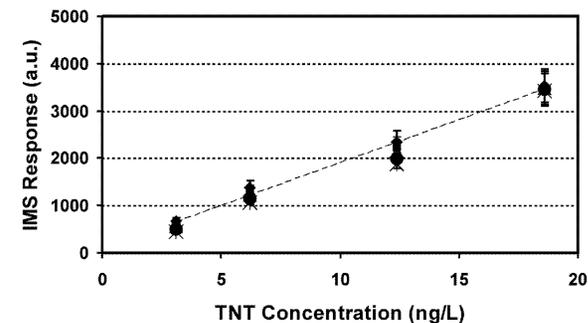
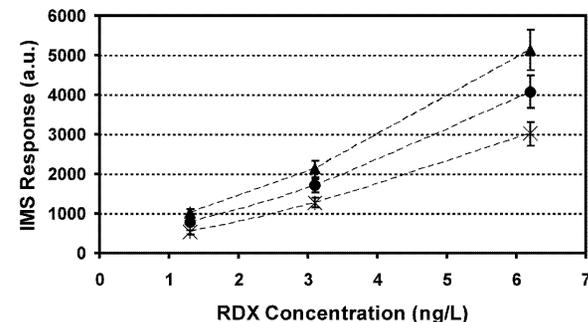
Compound ¹⁰	α (K ⁻¹)	β	$\Gamma_{130^\circ\text{C}} \times \text{Area}^*$ (ng/s)	Λ_{max} (ng/s)
RDX	-6473	16.50	2.5	1.0 [†]
PETN	-7243	19.56	42	4.2 [‡]
TNT	-5481	16.37	550	4.2 [‡]

* Droplet impingement area = 2700 μm^2

IMS Calibration Curves



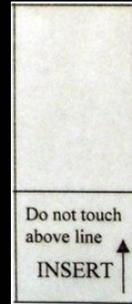
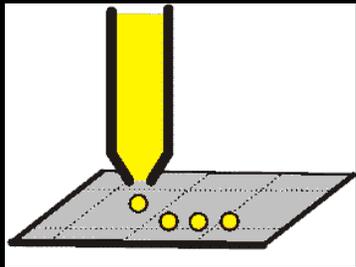
GE Security VaporTracer2
Single Vapor Mode w/ Preconcentrator
RDX, TNT, PETN



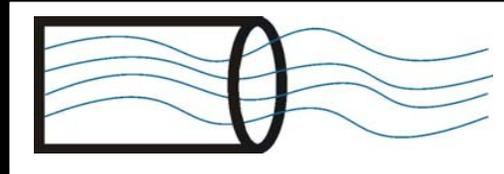
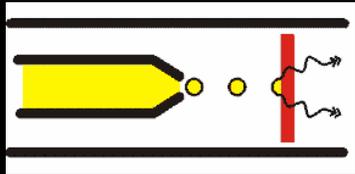
Legend for Sample Preconcentration Time:
 ◆ 50 sec ■ 40 sec ▲ 30 sec ● 20 sec * 10 sec

Summary

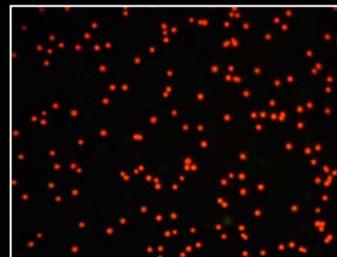
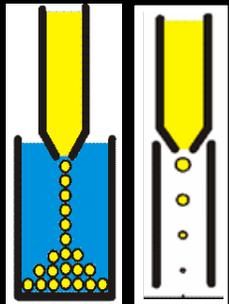
- Quant-Jet ... Printed Swipe Standards (IMs, TMs)



- Vapor-Jet ... Vapor Standards (Calibrations)



- Sphere-Jet & Particle-Jet... Portal Standards (TMs)





Advanced Measurement Laboratory, NIST, Gaithersburg, MD